PATENT
Docket No. Q4802

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

APPLICANTS:	Masaki Fukumori et al.	CONF.:	1178
APPLN. NO.:	10/579,217	ART UNIT:	1796
FILED:	May 12, 2006	EXAMINER:	NGUYEN, VU ANH
FOR:	AQUEOUS LIQUID DISPERSION OF WATER AND OIL REPELLENT AGENT CONTAINING NONIONIC SURFACTANT		

DECLARATION UNDER 37 C.F.R. § 1.132

Assistant Commissioner of Patents
Alexandria, VA 22313-1450

Sir:

I, Takashi Enomoto, residing in Osaka, Japan, do declare and say as follows:

1. THAT I am a citizen of Japan.

2. THAT I am a graduate of the Department of chemical for materials of Graduate School of Engineering, Mie University and received my master degree in 1986.

3. THAT I have been employed by DAIKIN INDUSTRIES LTD. since April 1, 1986, where I hold a position as researcher, with responsibility for research works on the development of the synthesis of fluorine-containing compound and the development of water and oil-repellent; and I worked in production department of fluorine-containing products between 1996 and 1999. Again, I started work for developing water and oil-repellent from 1999 to now. Especially, I worked in USA (DAIKIN AMERICA) between 2000 and 2006.

4. THAT I have read and understood the references cited in the Office Action notified on July 6, 2009.

5. THAT the following experiments were carried out by me or under my direct supervision and control.

6. Experiments

The experiments were carried out as follows:

[Evaluation]

Properties are determined as follows:

Storage Stability

The average particle diameter (scattering intensity) of an aqueous dispersion (solid content: 30% by weight) is measured at the initial and after the storage at 25°C for one month by FPAR-1000 manufactured by Otsuka Electronics Co., Ltd. and the storage stability is evaluated in the following criteria:

Good: Change rate of smaller than 10%
Fair: Change rate of 10% to 20%
Poor: Change rate of larger than 20%

Dilution Stability and Water- and Oil-repellency

(1) In the case of usual treatment

An aqueous dispersion is diluted with tap water to the solid content of 1.0% by weight, to prepare a treatment liquid. The state of the treatment liquid is observed after one hour, and the dilution stability is evaluated in the following criteria.

Good: No sedimentation
Fair: Slight sedimentation
Poor: Much sedimentation

After the evaluation of stability, a polyester cloth is immersed in the treatment liquid, squeezed with a mangle to give a wet pick up of 75%, dried at 100°C for 2 minutes, and heat-treated at 160°C for 1 minute, and then water- and oil-repellency of the treated cloth is evaluated.

Water repellency: AATCC-22 method
Oil repellency: AATCC-118 method

(2) In the case that acrylic binder (anionic) is used in combination

An aqueous dispersion is diluted with tap water to the solid content of 0.5% by weight, and an acrylic binder (ABCO Builder T-37 manufactured by ABCO) in the amount of 20.0% by weight is added to prepare a treatment liquid. The state of the treatment liquid is observed after one hour, and the dilution stability is evaluated in the following criteria.

Good: No sedimentation
Fair: Slight sedimentation
Poor: Much sedimentation

After the evaluation of stability, a polyester cloth is immersed in the treatment liquid, squeezed with a mangle to give a wet pick up of 110%, and heat-treated at 190°C for 2 minutes, and then water- and oil-repellency of the treated cloth is evaluated.

Water repellency: AATCC-22 method
Oil repellency: AATCC-118 method

(3) In the case that a fixing agent (cationic) for cotton is used in combination

An aqueous dispersion is diluted with tap water to the solid content of 1.0% by weight, and a fixing agent for cotton (NEOFIX RP-70 manufactured by Nicca Chemical Co., Ltd.) in the amount of 0.5 % weight is added to prepare a treatment liquid. The state of the treatment liquid is observed after one hour, and the dilution stability is evaluated in the following criteria.

Good: No sedimentation
Fair: Slight sedimentation
Poor: Much sedimentation

After the evaluation of stability, a polyester cloth is immersed in the treatment liquid, squeezed with a mangle to give a wet pick up of 60%, and heat-treated at 160°C for 1 minute, and then water- and oil-repellency of the treated cloth is evaluated.

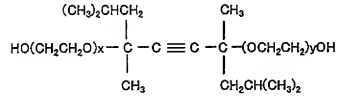
Water repellency: AATCC-22 method
Oil repellency: AATCC-118 method

[Test]

A test was carried out wherein nonionic surfactants that were employed in Example 1 in Oharu et al. (JP 2001-107031 A; hereinafter Oharu) were used as the nonionic surfactants having of HLB of less than 12. Specifically,

Surfactant A: A polypropylene oxide/polyethylene oxide block copolymer of which an average molecular weight of a polypropylene oxide portion was 2000 and of which average addition mol number pf ethylene oxide of a polyethylene oxide portion was 30; and

Surfactant B: A compound represented by a following formula:



average sum of x and y : 1.3

were prepared as the nonionic surfactants having of HLB of less than 12. Surfactant A had an HLB of 11.8 (calculated by Griffin method) and surfactant B had an HLB of 4.

Next, an aqueous water- and oil-repellent dispersion containing a polymer was prepared following the same procedures described in Example 1 of the specification of the present application (see pages 20-21) except that surfactant A (2 g) and surfactant B (4 g) were replaced with surfactant 1 (6 g), the ratio of surfactant A to surfactant B being 1:2 which is the same as that employed in Example 1 of Oharu. Specifically, into a 1 L autoclave, $C_nF_{2n+1}CH_2CH_2OCOCH=CH_2$ (a mixture of compounds wherein n is 6, 8, 10, 12 and 14 (average of n: 8)) (FA) (a fluorine-containing monomer) (200 g), stearyl acrylate (20 g), N-methylolacrylamide (3 g), tripropylene glycol (50 g), pure water (400 g), surfactant A (2 g) and surfactant B (4 g), nonionic surfactant 3 (16 g), nonionic surfactant 5 (4 g) were charged and emulsified by ultrasonic wave at 40°C for 30 minutes with stirring. After the emulsification, n-dodecyl mercaptan (1 g) was added and then vinyl chloride (40 g) was injected. Further, azobisisobutylamidine dihydrochloride (0.8 g) was added and the reaction was conducted at 60°C for 5 hours to give the aqueous water- and oil-repellent dispersion containing the polymer. The composition of the polymer was that the reaction conversion of vinyl chloride was about 80% and the reaction conversions of the other monomers were about 100%.

Then, for the resultant aqueous water- and oil-repellent dispersion, evaluated were storage stability; water- and oil-repellency and stability of the treatment liquid in the case of usual treatment; water- and oil-repellency and stability of the treatment liquid in the case that an acrylic binder (anionic) is used in combination; water- and oil-repellency and stability of the treatment liquid in the case

that a fixing agent (cationic) for cotton is used in combination. The results are shown in the following Table I. For information, the evaluation results of Examples 1-7 shown in the specification of the present application are shown again in this table.

Table I

	Nonionic Surfactant			Ionic Surfactant	Storage Stability	Usual Treatment			Use of Acrylic binder			Use of Fixing agent		
						Polyester fabric			Polyester non-woven fabric			Cotton fabric		
	HLB: less than 12	HLB: at least 12 and less than 17	HLB: at least 17			Water repellency	Oil repellency	Stability	Water repellency	Oil repellency	Stability	Water repellency	Oil repellency	Stability
Test	A and B	3	5		Fair	4	5	Good	4	3	Fair	4	5	Good
Ex. 1	1	3	5		Good	5	6	Good	5	4	Good	5	6	Good
Ex. 2	1	4	6		Good	5	5	Good	5	5	Good	5	5	Good
Ex. 3	2	3	5		Good	5	4	Good	4	4	Good	4	4	Good
Ex. 4	2	4	6		Good	4	4	Good	3	3	Good	4	3	Good
Ex. 5	1	3	5	A(5)	Good	5	6	Good	4	4	Good	5	6	Good
Ex. 6	1	3	6	B(2)	Good	4	4	Good	4	4	Good	4	4	Good
Ex. 7	2	4	5	C(10)	Good	4	3	Good	4	3	Good	3	3	Good

In Table I, the numbers shown in the columns of "Nonionic Surfactant" and "Ionic surfactant" indicate the following compounds shown in Table II and Table III.

Table II

Nonionic surfactant

		HLB
1	$C_{11\text{-}14}H_{23\text{-}29}$-iso-O$(C_2H_4O)_3H$	8.0
2	$C_{12}H_{25}O(C_2H_4O)_4H$	9.2
3	$C_{11\text{-}14}H_{23\text{-}29}$-iso-O-$(C_3H_6O)_3(C_2H_4O)_{20}H$	14.0
4	$C_{12}H_{25}O(C_2H_4O)_{20}H$	16.2
5	$C_{12}H_{25}O(C_2H_4O)_{30}H$	17.3
6	C_8H_{17}-  -O-$(C_2H_4O)_{40}H$	17.9

Table III

Ionic surfactant

		Ionicity
A	Lauryl trimethyl ammonium chloride	Cationic
B	Sodium lauryl sulfate	Anionic
C	Lauryl dimethyl aminoacetate betaine	Amphoteric

7. Results

As apparent from the table, the aqueous water- and oil-repellent dispersion obtained in this test were inferior in storage stability compared to the dispersions (Examples 1-7) wherein $C_{11\text{-}14}H_{23\text{-}29}$-iso-$O(C_2H_4O)_3H$ (HLB value:8.0) or $C_{12}H_{25}O(C_2H_4O)_4H$ was used as the nonionic surfactant having HLB of less than 12, even though surfactants A and B have HLB of less than 12 and they were used in combination with the surfactant having HLB of not less than 12 and less than 17 and the surfactant having HLB of not less than 17.

8. The undersigned petitioner declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

9. Further deponent saith not.

Date: Nov. 27/2009 By Takashi Enomoto

Takashi Enomoto